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WORKING STEEL P2-04BCH BY EQUAL CHANNEL ANGULAR EXTRUSION (ECAE)

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The article presents results of investigation of structure and properties of low-carbon steel grade P2-04BCH after application of ECAE at temperature of approx. 290 °C. The ECAE method leads to significant improvement of strength of investigated material. Investigation of structure was made by combination of TEM and FEG SEM together with EBSD. It was proven the ECAE method enables obtaining of ultra fine-grained ferritic structure formed by recrystallised grains with very low density of dislocations and with a small portion of spheroidised carbides, which occurred usually on the boundaries of ferritic grains.

Key words: nanomaterials, low-carbon steel, ECAE deformation, EBSD

Obrada čelika P2-04BCH kutno kanalnom ekstruzijom (KKE). Članak prikazuje rezultate istraživanja strukture i svojstava niskougljičnog čelika kvalitete P2-04BCH nakon kanalno kutne ekstruzije (KKE) pri temperaturi od oko 290 °C. Zabilježen je značajan porast čvrstoće materijala. Struktura je ispitana kombinacijom transmisijskog elektronskog mikroskopa (TEM), skenirajućeg elektronskog mikroskopa s emisijom polja (FEG SEM) te elektronskom difrakcijom (EBSD). Dokazano je da se jednakokanalnom kutnom ekstruzijom postiže izuzetno sitnozrnata feritna struktura rekristalizacijom zrna, uz vrlo nisku gustoću dislokacija i mali udio sferičnih karbida s rasporedom, obično na granicama feritnih zrna.

Ključne riječi: nanomaterijali, niskougljični čelik, deformiranje jednakokanalnom kutnom ekstruzijom, povratno rasipajuća elektronska difrakcija

INTRODUCTION

Enhancement of strength properties of polycrystalline metallic materials with preservation of sufficient toughness can be achieved by refining of grains [1, 2]. Dependence between the grain size and the level of yield strength is described by the Petch–Hall relation

$$\sigma_y = \sigma_0 + k \cdot d^{-1/2} \qquad (1)$$

where σ_y is flow stress, σ_0 and k are constants, d is grain size.

This relation can be used in extensive interval of grain sizes, up to several tens of nanometres [3]. The searching of possibilities of efficient grain-refinement of structure of technical materials led to important modifications of technology of thermo-mechanical treatment, which enable obtaining grain size at the level of several micrometres.

The most efficient processes are the following: deformation induced ferritic transformation, dynamic re-crystallisation of austenite during hot deformation with subsequent $\gamma \rightarrow \alpha$ transformation, hot rolling at inter-critical interval of temperatures and dynamic recrystallisation of ferrite after large hot deformation [3].

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Further refining of grain size requires, however, application of extreme value of plastic deformation of material. During last two decennia many methods were developed that enable achievement of severe plastic deformation. Important position among them holds the equal channel angular extrusion (ECAE) [4-6]. Principle of this method consists of severe deformations of massive samples realised by shear without change of cross section. The sample is pressed through a die, in which two channels intersect, forming an angle usually of 90°. Pressing is made either at room or at increased temperature. Equivalent deformation can achieve the value of 10 or even higher.

The most critical for development of microstructure and resulting properties of samples is above all number of passes and selection of deformation route (manner of turning of the sample after each pass). It was established from the analysis of shear characteristics at various deformation routes that turning of the sample by 90° was optimal. Many works, dealing with optimisation of the laboratory ECAE equipment, were published. Promising modifications for production of ultra fine-grained massive semi-products in industrial practice have appeared [3, 4, 6, 7].

The ECAE method makes it possible to obtain the grain size of several hundreds of nanometres [8-11]. Materials with sub-micron size of sub-grains/grains ($d =$

0,1-1 μm) are usually classified as ultra fine-grained materials [3].

The ECAE method was so far unsuccessful at attempts of obtaining nanometric materials, i.e. materials with grain size under 0,1 μm. Characterisation of the fraction of sub-grains formed by recovery and grains separated by high-angle boundaries, which are formed by re-crystallisation [11], is very important for understanding the mechanisms of materials structure evolution at application of methods of extreme plastic deformation. Definition of difference between sub-grains and grains is not rigid. The values of 10-15° [1, 2] are usually given as a critical misorientation angle. It is known that grains separated by high-angle boundaries have generally much more important influence on the level of mechanical properties than sub-grains divided by low-angle boundaries [2]. In the area of grain size under approx. 0,3 μm the classical mechanism of plastic deformation by dislocation sliding is replaced by other mechanisms. The most important causes of this phenomenon comprise increasing surface of grain boundaries per unit of volume of material, decrease of dislocation density inside the grains with grain size under 0,1 μm, and localisation of deformation into shear bands. Important problems connected with development of ultra fine-grained materials include in the first place lower level of plasticity non-homogeneity of structure across cross section of the pressed blanks and thermal stability of ultra fine-grained structure at higher temperatures [3].

Majority of works published until now dealt with application of the ECAE method on pure metals, while much less attention was given to investigation of commercial steels [10]. Our article summarises the results obtained at investigation of severe plastic deformation by the ECAE method on strength characteristics and structure of a low-carbon steel P2-04BCH. Detailed investigation of structure evolution was made not only with use of Transmission Electron Microscopy (TEM), but also by Scanning Electron Microscopy (SEM) in combination with Electron Back Scattered Diffraction (EBSD) [12], which enables characterisation of misorientation angles of individual crystallites on the surface of metallographic sections. At present if the Field Emission Gun (FEG) is used, it is possible to obtain the spatial resolution of approx. 0,1μm.

Experimental material and technique

Investigation was made with the use of a commercial low-carbon steel grade P2-04BCH. Table 1 gives its chemical composition.

Table 1. **Chemical composition of the steel P2-04BCH / mas. %**

C	Mn	Si	Cr	Mo	Ti	B
0,034	0,67	0,23	0,10	0,017	0,001	0,002

The material supplied was in the state after free air cooling from the rolling temperature. Cylindrical samples of dimensions ϕ12×60 mm were manufactured from this initial material. The angle between the channels of the used ECAE die was 105°. This design made possible to reduce deformation resistance and it ensured good filling of die edges [13]. The samples were before the pressing re-heated in the furnace to temperature of approx. 320 °C, temperature of the ECAE die was approx. 290 °C. Deformation route B_c was applied (turning of the sample after each pass by 90° in the same direction), moreover the front end of the sample was replaced by the rear end of the sample. This deformation route is considered generally as the quickest manner of achievement of homogenous structure formed by equiaxed grains [14]. The maximum number of realised passes through the ECAE die was 16.

Samples for tensile test were prepared from individual deformed samples. This test was made at room temperature. For the purposes of structural analysis sections were prepared perpendicularly to the longitudinal axis of the samples after 4 (equivalent deformation ε = 3,5) and 8 (ε = 7,1) passes through the ECAE die. Final polishing of the samples for the SEM analysis was made with use of colloidal solution of SiO_2 with granularity 0,05 μm. Crystal orientation maps (COM), study on misorientation angles of individual sub-grains/grains and statistic evaluation of grain size was made by the apparatus Sirion 200 FEG SEM equipped with the HKL Technology Channel 5 EBSD system. Thin foils for the TEM were prepared perpendicularly to the longitudinal axis of the samples from the approximately ¼ of the diameter of initial samples. The foils were prepared by electrolytic polishing in the solution containing 5 % of $HClO_4$ and 95 % of CH_3COOH at the room temperature and voltage of 60 V. The TEM investigation was performed on the microscope JEOL JEM 2100 equipped with the PGT EDX analyser.

EXPERIMENTAL RESULTS AND DISCUSSION

Microstructure and mechanical properties of steel in the initial state

Results of the tensile test of the supplied material at the room temperature are given in Table 2.

Table 2. **Results of tensile test of the steel P2-04BCH**

$R_{p0,2}$ / MPa	R_m / MPa	A / %	Z / %
281	355	31,5	72,5

Microstructure of steel was formed by equiaxed grains of ferrite, which were discontinuously decorated by carbidic particles, see Figure 1. Small islands of decomposed ferritic-carbidic component were present in a very small quantity at the boundaries of ferritic grains. Small precipitates were observed also inside ferritic grains. Average size of ferritic grains was approx. 35 μm.

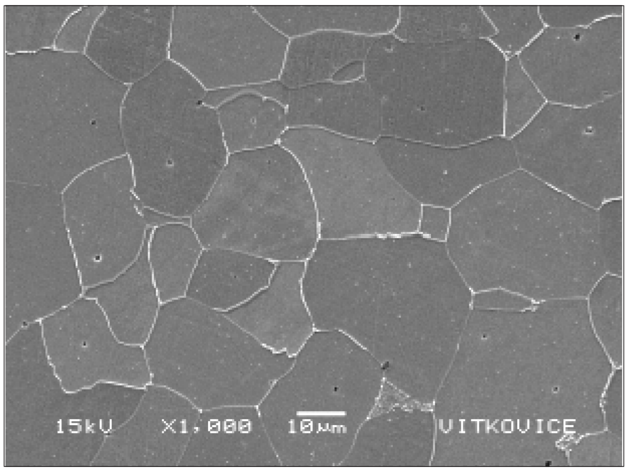

Figure 1. Microstructure of steel in the initial state

Microstructure and mechanical properties of steel after application of ECAE

Severe plastic deformation of the investigated steel in the ECAE die lead to significant enhancement of strength properties. Obtained results are shown in Figure 2.

The biggest increase in strength properties was found after the first two passes. Next passes resulted only in very gradual enhancement of strength parameters. After 16 passes even slight decrease of strength properties was already observed.

Microstructure of the steel investigated after 4 ECAE passes was non-homogeneous, original ferritic grains were largely deformed. Deformed ferritic grains formed in elongated bands are clearly visible in Figure 3.

TEM analysis proved the original equiaxed ferritic grains were replaced by stretched sub-grains/grains of variable size. Sub-grains/grains formed usually elongated parallel bands, see Figure 4.

EBSD results obtained on the sample after 8 ECAE passes were processed in the form of crystal orientation maps (COM), where the areas of various orientations on the sample surface are discriminated by different colouring. The obtained results were further processed by computer as follows:

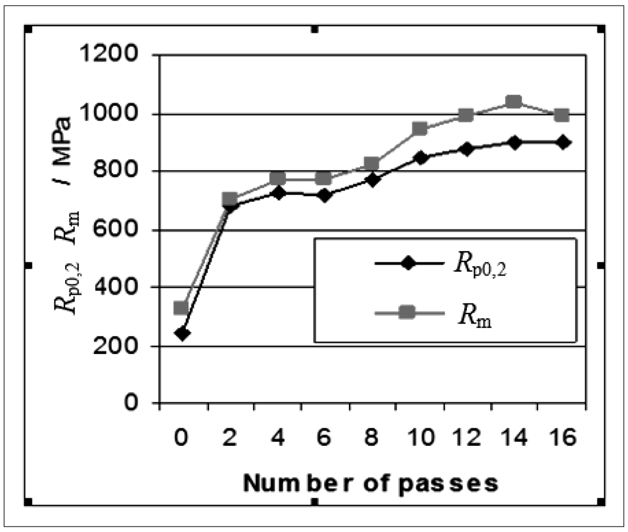

Figure 2. Results of tensile tests of material deformed in the ECAE die

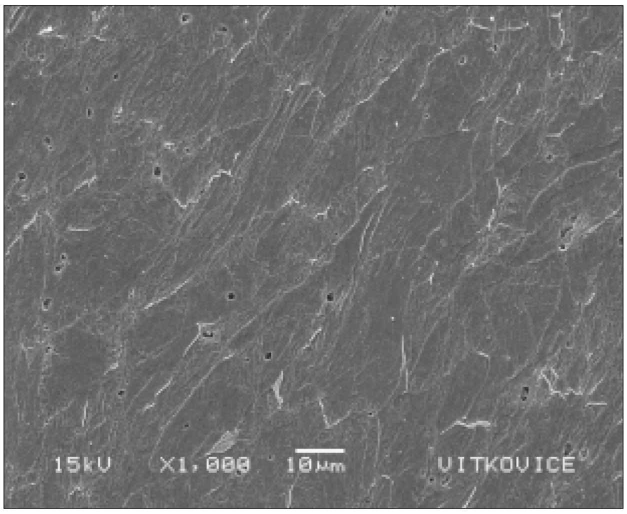

Figure 3. Microstructure of the sample after 4 ECAE passes

- in the areas, where the misorientation angle of adjacent pixels was greater than 2°, the boundaries were plotted. In this manner boundaries of sub-grains were visualised, as well as boundaries of grains separated by high-angle boundaries.
- in order to differentiate between the sub-grains and grains the boundaries of grains were plotted only in the areas, where misorientation of adjacent pixels exceeded 10°.
- grain boundaries were plotted in the areas, where misorientation of adjacent pixels exceeded 20°.

Map of crystal orientations (COM), shown in Figure 5, documents a large quantity of differently oriented sub-grains/grains in the investigated area.

Results of statistic processing of misorientation angles of sub-grains and grains in the sample after 8 ECAE passes are shown in Figure 6. It is obvious that sub-grains with the misorientation angle under 10° formed only in approx. 15 % of all ferritic grains. This confirms the fact the majority of ferritic grains was formed by the mechanism of re-crystallisation. No preferential occurrence of special boundaries was observed in the area of high-angle boundaries, such as e.g. twin

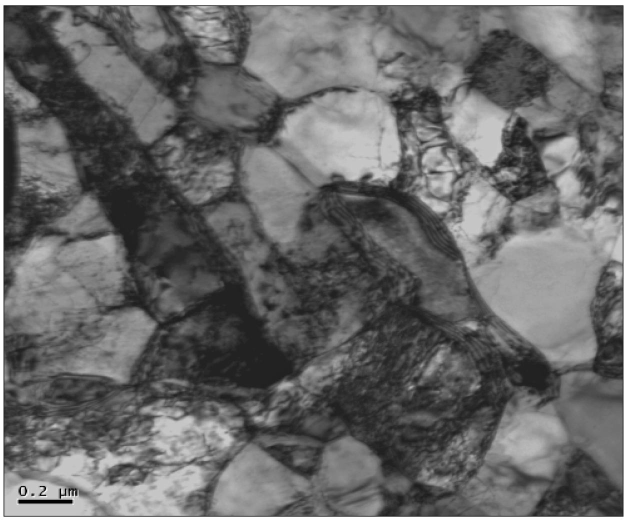

Figure 4. Substructure of the sample after 4 ECAE passes

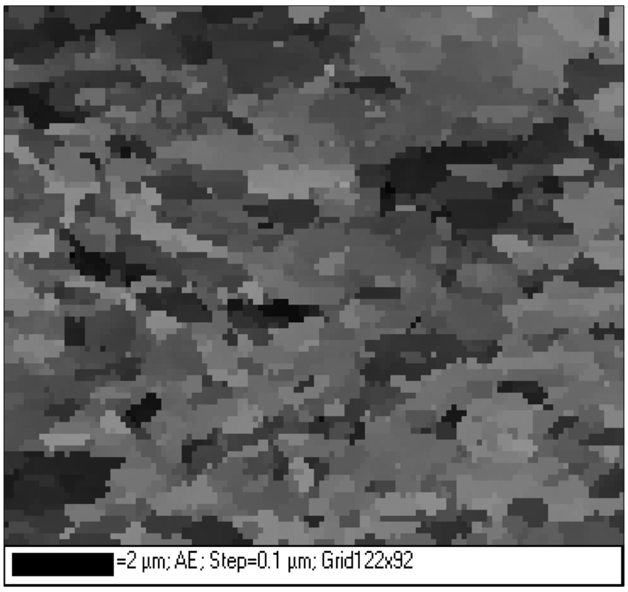

Figure 5. Crystal orientation map, sample after 8 ECAE passes

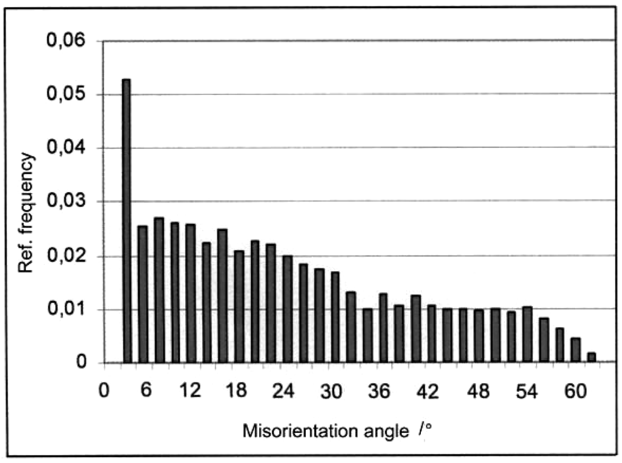

Figure 6. Distribution of misorientation angles of sub-grain and grain boundaries, sample after 8 ECAE passes

boundaries [15-17]. The biggest share of sub-grains corresponded to the misorientation angles up to 4°.

CONCLUSIONS

The results obtained from analysis of influence of severe plastic deformation by the ECAE method on structure and properties of the low-carbon steel grade P2-04BCH can be summarised as follows:

- Deformation of investigated steel by the ECAE method at the temperature of approx. 290 °C led to significant improvement of strength properties. The decisive increase in strength was found after the first two passes through the ECAE die.
- Deformation applied in 8 ECAE passes led to formation of ultra fine-grained ferritic structure with a small fraction of globular carbidic particles, which were usually present at the boundaries of ferritic grains. Density of dislocations inside ferritic grains was very low. Majority of ferritic grains was formed by the mechanism of re-crystallisation of deformed metallic matrix.
- Sub-grains with an angle of misorientation under 10° were formed after 8 ECAE passes only in approximately 15 % of all ferritic grains.
- Average size of ferritic grains with high-angle boundaries after 8 ECAE passes was 0,32±0,20 μm. However, the analysis could not include the grains, the size of which was smaller than 0,1 μm. In comparison with the as received state the grain size was refined by two orders.

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